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AGING OF LARGE SIZE FRP/EPOXY ADHESIVE BONDS AGED UNDER WATER AND VACUUM - INTERIM REPORT FOLLOWING NINE MONTHS AGING

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ABSTRACT

Tensile test specimens of epoxy adhesive bonded FRP with three different bond areas and six different area to bond line exposed to water were designed and manufactured and then aged in wet vacuum conditions for periods of up to nine months. The unaged specimens exhibited a lower initial bond strength than in results from a previous test program. Unaged tensile bond strength was found to decrease with increasing bond area to an asymptotic value and the manufacture and testing of unaged specimens of three more different bond areas confirmed this phenomenon. Although all the specimens tested so far indicate an apparent bond strength degradation over the aging period, the data is insufficient at this time to determine the degree of degradation.

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32-167-453	835.5	November 30, 1990	90-92-K

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EXECUTIVE SUMMARY

AGING OF LARGE SIZE FRP/EPOXY ADHESIVE BONDS AGED UNDER WATER AND VACUUM - INTERIM REPORT FOLLOWING NINE MONTHS AGING

R.J. Lewak

Prior to the 1980 station outage at Pickering NGS, the Atomic Energy Control Board (AECB) had raised concerns about the structural integrity of the Fiberglass Reinforced Plastics (FRP) pipes used in the Dousing system. As a result, FRP stiffening rings were installed on both risers and downcomers to increase safety margins against collapse under a full LOCA load. Each six part stiffening ring was comprised of an upper and lower half, each half divided into three 120° segments for installation. As well as being bonded to the riser or downcomer using an epoxy adhesive, each of the segments were bonded to the adjacent segments and the opposite half of the ring in such a manner that the adhesive lines between the upper half is offset from those of the lower half. A previous study using small bond area FRP/epoxy adhesive specimens with varying thickness of epoxy adhesive bond aged under the vacuum building conditions of vacuum and water indicated that the tensile bond strength might deteriorate with aging time. It is believed that this deterioration was caused by water ingress into the bond around the bond perimeter. In the previous study the ratio of bond perimeter exposed to water to bond area was constant for all specimens, there was no data on the relationship of this ratio to bond degradation. The previous study also had a small bond area giving a small bond perimeter to area ratio which was in no way comparable to the actual bonds in the Pickering NGS vacuum building. The test program in the previous study had a two year duration which can be extrapolated to a period of twenty years. Since the vacuum building is expected to be in service beyond the year 2000 additional testing over a longer test period was

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foreseen to be required. Therefore, a new study with varying bond areas and varying perimeter to bond area ratios was conceived and implemented.

This current study involves the design, manufacture and testing of three different tensile bond area FRP/Epoxy specimens to determine the effect of aging under water and vacuum for a time period of up to 144 months. At this point in time, ten specimens each of bond areas of 12.9 cm^2, 3.2 cm^2, and 0.81 cm^2 were tensile tested after aging periods of 0, 3, 6, and 9 months. Additionally, the perimeter of the bond area which is open to water ingress is also varied within each type of bond area so that the ratio of exposed perimeter to area could also be studied for effects of aging. Initial test results showed a decrease to an asymptotic value in bond strength with bond area for the unaged specimens. The unaged results also showed a lower bond strength than in the previous test program which could be accounted for by different specimen preparation and formulation changes in the epoxy adhesive. It was concluded that while the previous test program could be used for determination of the absolute bond strength between the FRP and the epoxy adhesive in the Pickering vacuum building, because the adhesive formulation and bond fabrication was more similar to the VB conditions, the results of the present study could be used for trend indication with regard to bond area variations in strength and ingress perimeter to bond area variations strength with aging time.

Three month aged specimens generally showed a decrease in bond strength. These results were discounted, however, as the ratio of exposed bond perimeter to bond area was too large for each bond area type to be at all representative of actual vacuum building bond conditions. By sealing the perimeter of the bonds with vacuum grease to prevent water infiltration but leaving a fraction of the bond perimeter open to infiltration the aging condition became more representative.

After six months aging there seemed to be an apparent decrease in bond strength. Sufficient data, however, was not available to evaluate the decrease by statistical analysis. The nine month aged specimens also showed an apparent decrease in bond strength from the unaged specimens. However, there was still not sufficient data to evaluate the decrease and to gauge its significance. It is recommended that this project continue until completion to a 144 month aging period, with testing at times of 12, 24, 48, 72, 96 and 144 months, in order that trends in bond strength degradation be established over a representative period of time.

It has also been recommended that a parallel test program be instituted on bond shear strength using varying bond areas over a similar time period and schedule. This test program would be

effective in that although the previous test program indicated that the decrease in shear strength of the epoxy bonded FRP specimens was insignificant, the previous program was carried out over only a two year period which could be extrapolated to 20 years or to the year 2000. Since the station life is expected to last beyond 2000, it would be advantageous to confirm that the decrease in shear strength of the bonds over a longer time period is not significant.

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To: Mrs. N. Yousef
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AGING OF LARGE SIZE FRP/EPOXY ADHESIVE BONDS AGED UNDER WATER AND VACUUM - INTERIM REPORT FOLLOWING NINE MONTHS AGING

1.0 INTRODUCTION

Prior to the 1980 station outage at Pickering NGS, the Atomic Energy Control Board (AECB) had raised concerns about the structural integrity of the Fiberglass Reinforced Plastic (FRP) pipes used in the Dousing system. As a result, FRP stiffening rings were installed on both risers and downcomers to increase safety margins against collapse under all postulated accident conditions. Each stiffening ring had a top and bottom half each divided into three sections, each section comprising 120^{o} of arc. Each section was bonded to its riser or downcomer and to its respective neighbouring sections by an epoxy resin adhesive. This 'Epoxy F' adhesive was supplied by Fibergrid Inc.(formerly Protective Plastics Ltd. of Scarborough, Ontario) who manufactured the FRP pipes for Pickering NGS dousing system.

A research program was undertaken by the Chemical Research Department to determine the effects of wet vacuum conditions normal to the Vacuum building on the bond strength between FRP and Epoxy 'F'. Tensile and shear specimens were made according ASTM D2095 (Tensile Strength of Adhesives by Means of Bar and Rod Specimens) and ASTM D3165 (Strength Properties of Adhesives in Shear by Tension Loading of Laminated Assemblies) and were aged under water and near vacuum and then tested for bond strength /1/. These tests were performed on unaged specimens and specimens aged for 3, 6, 12, and 24 months. The thickness of the epoxy between the FRP was varied in this test but not the bond area. The results of this program showed that there was no change in the bond strength of the shear specimens over a 24 month aging period. However, the results also showed that there

was a significant degradation in the bond strength of the tensile specimens. Varying the thickness of the epoxy in the bond did not change these trends. The bond area of the tensile specimens was only 1.6 cm^2. However, the actual bond area between two arc sections of each ring, where the tensile stress on the bond is the greatest under postulated accident conditions, is approximately 9 cm^2. Because the specimens had their entire perimeter exposed to the water, a greater percentage of the total bond area was liable to water ingress than is the situation with the stiffening ring's bond to the risers and downcomers. It was felt that the results were at best quite conservative and at worst they may not be directly applicable to predicting the degradation of bond strength between the stiffening rings and risers or downcomers.

In order to determine if the degradation rate of larger bond areas is similar to that of the small specimens, if there is a relation between bond degradation and the ratio of bond area to circumference, and whether the degradation rate reaches a limiting value over a time period, the current program was undertaken. In this program tensile bond strength specimens of FRP and Epoxy "F" were manufactured with three different bond areas, two of which are significantly larger than the bond area of the previous test program. The specimens were aged under vacuum and water conditions and then tensile tested after aging periods of 0, 3, 6, and 9 months up to this point in time.

2.0 SPECIMEN DESIGN AND FABRICATION

2.1 Specimen Design

The design of the specimen for the tensile testing of bond strength of large area FRP/Epoxy bonds formed a major portion of the test program. The problems involved were basically threefold:

1. The design of the specimen had to be such that the maximum stresses occur in the FRP/Epoxy bond interface during the tensile test.

2. In certain instances the bond strength between the FRP and epoxy may be greater than the bond strength between FRP laminae. Therefore, the specimen must be designed so that the tensile stress is not transmitted from the FRP/Epoxy bond to the FRP during the tensile tests.

3. Because this test program investigates long term aging effects, specimens would be tested after aging periods of 0, 3, 6, 9, 12, 24, 48, 72, 96, and 144 months. For statistical significance at least 10 specimens of each of three bond areas are required for testing which results in a total

of 300 specimens, 100 of each bond area. The manufacture of 300 specimens in a reasonable period of time requires that mass production techniques must be used. Therefore the specimen design must be suitable for mass production methods.

To solve the first problem, the basic shape of the tensile specimen was chosen to be a common dumbbell shape with the two large ends of the dumbbell to be made from FRP and the narrow portion to be made from Epoxy "F". The second problem could be solved by using, instead of conventional tensile test grips, a set of grips which would pull the specimen from beneath the FRP so that a compressive force would be applied to the FRP while a tensile force would be applied to the FRP/Epoxy bond. A set of grips of this type was designed and manufactured and are shown in Figure 1 holding a specimen. The grip is essentially a steel box made to attach onto the pull rods of a tensile machine by the threaded fitting on the top plate. The specimen, which is to be two FRP blocks joined together by an Epoxy ligature, fits into the open space so that the FRP block is supported from below and the epoxy ligature goes through the opening between the bottom arms.

The solution to the third problem completes the design of the specimens. For mass production methods, the specimen must be kept as simple as possible. The simplest design is to have two blocks of FRP bonded together using three different areas of epoxy. From the previous test results on tensile specimens having a bond area of 1.6 cm^2 (0.25 in^2) the maximum bond strength was 37.2 MPa (5400 psi). Assuming a linear relationship between bond strength and bond area, a bond strength of 37.2 MPa would allow for a maximum bond area of 13.44 cm^2 using a 50 kN load cell. It was thought that a 50 kN load cell was the largest which could be used while retaining a reasonable load resolution. For easier manufacture the maximum bond area was set at 12.9 cm^2 (2 in^2). The other two bond areas were chosen as 1/16 and 1/4 of the maximum bond area to be 0.81 cm^2 (0.125 in^2) designated as Type A and 3.2 cm^2 (0.5 in^2) designated as Type B while the largest bond area is Type C. Because the maximum load could be 37.2 MPa the FRP blocks must be of sufficient thickness to prevent bending and thus placing shear stresses on the FRP/Epoxy bond. A thickness of 2.54 cm (1 in) was chosen for this thickness. The length and width of the FRP blocks were chosen as 7.62 x 5.08 cm. (3 x 2 in). To apply the load over a large area on the underside of the FRP blocks the load applying support arms of the grips were 2.54 x 7.62 cm. (1 x 3 in) and required a thickness of 0.95 cm (0.375 in). Therefore, to provide clearance for both grips the epoxy ligature between FRP blocks was kept at a constant 2.54 cm. for all specimens. The three bond areas were:

Type A: 0.635 cm along the length and 1.27 cm along the width of the block (0.25 x 0.5 in.)
Area = 0.81 cm^2 (0.125 in^2)
Bond perimeter = 3.81 cm (1.5 in.)
Ratio of area to perimeter = 1:4.72 cm (1:12 in.)

Type B: 2.54 cm along the length and 1.27 cm along the width of the block (1 x 0.5 in.)
Area = 3.2 cm^2 (0.5 in^2)
Bond perimeter = 7.62 cm (3 in.)
Ratio of area to perimeter = 1:2.36 cm (1:6 in.)

Type C: 2.54 cm along the length and 5.08 cm along the width of the block (1 x 2 in.)
Area = 12.9 cm^2 (2 in^2)
Bond perimeter = 15.24 cm (6 in.)
Ratio of area to perimeter = 1:1.2 cm (1:3 in.)

2.2 Specimen Manufacture

The FRP was ordered in 25 30.48 cm x 30.48 cm x 2.54 cm thick sheets from Fibergrid Inc. (formerly Protective Plastics Ltd.) of Scarborough, Ontario, the original suppliers of the FRP in the Pickering NGS vacuum building. The original layup was used with:

1 Layer M514 ('C' type glass veil, Owens Corning)
2 Layers M751 ('E' equivalent type glass, chopped strand mat, 1.5 oz./ft^2, Fiberglass Canada)
1 Balanced Layup of glass fiber cross-ply (65-70% glass)
2 Layers M751
1 Layer M514

The resin used in the FRP is:

ATLAC 382-05, 50% styrenated Bisphenol A Polyester	100 parts by weight
Dimethylaniline	0.1 parts by weight
6% Cobalt Napthenate	0.75 parts by weight
Methyl Ethyl Ketone Peroxide	1.0 parts by weight

The epoxy adhesive, Epoxy "F", was supplied by Fibergrid, Inc. in 1 liter containers and had a formulation as:

Epoxy Resin	100 parts by weight
Inorganic Mineral Fillers	100 parts by weight
Triethylenetetramine, Union Carbide	24 parts by weight

The FRP was cut and machined into 600 5.08 x 7.62 x 2.54 cm blocks in order to make 300 specimens. In order to duplicate the bonding procedure used in the vacuum building, the blocks all had the bond surfaces sanded to destroy the gloss of the resin.

As the Epoxy "F" was a viscous liquid before curing, in order to make the bond for each specimen with an epoxy ligature of 2.54 cm, the epoxy had to be cast in a mold. A total of thirty molds were made from aluminum as seen in Figure 2. The specimen fabrication procedure consisted of inserting two FRP blocks into the mold, filling the empty space with Teflon spacers so that the volume remaining would be either 2.54 x 0.635 x 1.27 cm for Type A specimens, 2.54 x 2.54 x 1.27 cm for Type B specimens, or 2.54 x 2.54 x 5.08 cm for Type C specimens, filling the mold with mixed Epoxy "F", allowing the epoxy adhesive to cure for 24 hours and then removing the specimen from the mold. This procedure was repeated until 100 of each type of specimen was fabricated. Type A specimen is shown in Figure 3, Type B in Figure 4, and Type C in Figure 5.

After the specimens had been aged for approximately 3 months, it was realized that the problem of water ingress on a small bond perimeter degrading an unrepresentative percentage of the bond area was present. To rectify this problem the specimens were removed from their aging tanks and the bond perimeters were sealed using vacuum grease. They were sealed in the following manner:

Type A: half with one length of 0.635 cm exposed, half with one length of 1.27 cm exposed on each bond.

Type B: half with one length of 2.54 cm exposed, half with one length of 1.27 cm exposed on each bond.

Type C: half with one length of 2.54 cm exposed, half with one length of 5.08 cm exposed on each bond.

This reduced the bond area to perimeter ratios to:

Type A: 1:0.79 cm (1:2 in) and 1:1.58 cm (1:4 in)

Type B: 1:0.39 cm (1:1 in) and 1:0.79 cm (1:2 in)

Type C: 1:0.2 cm (1:0.5 in) and 1:0.39 cm (1:1 in)

3.0 AGING CHAMBER DESIGN AND MANUFACTURE

The design of the aging chamber was based on the following specifications:

1. Interior conditions of vacuum building water and pressure of 6.89 kPa absolute

2. Space enough for 300 7.62 x 7.62 x 5.08 cm specimens.

3. Ease of removal of specimens without draining chamber.

The design of the aging chamber to meet these specifications is shown in Figure 6. Two chambers are required for 300 specimens. Each chamber is a 45.72 cm OD, 1.27 cm wall thickness, 60.96 cm long 316 stainless steel pipe to which a 1.27 cm thick stainless steel bottom and a 53.34 cm OD316 stainless steel flange has been welded. The matching top flange is bolted to the assembly by 18 1.27 cm hex bolts. A 0.635 cm diameter line is attached to the top flange which is connected to a vacuum pump. The pump is set on a timer to operate twice a day to reduce the pressure in the chambers to 6.89 kPa. The chambers were filled with Pickering NGS vacuum building water and the specimens to be aged on April 4, 1989.

4.0 TEST RESULTS FROM UNAGED SPECIMENS

A total of 30 unaged specimens, 10 of each type, were tensile tested to destruction using a Satec tensile testing machine for Type B and C specimens as shown in Figure 7 and a Baldwin tensile testing machine for the Type A specimens as shown in Figure 8 and the test grips designed for this project. Figure 9 shows a Type A specimen undergoing a tensile test procedure on the Baldwin machine while Figure 10 shows a Type C specimen undergoing a tensile test on the Satec machine. The strain rate was 1.0 mm/minute. All the specimens broke at the FRP/Epoxy bond similar to that shown in Figure 11. The test results for Type A, B, and C specimens are given in Tables 1, 2, and 3 respectively.

The test results show a considerable decrease in bond strength from that obtained in the previous test program/1/. In the previous test program the unaged bond strength for the single bond area of 1.6 cm^2 was 33 MPa. In this program the largest bond strength is 14.9 MPa. There are three possible reasons for this decrease. The first is that the pressure applied during specimen manufacture was no doubt different in the two test programs. The first test program, as in the vacuum building, had a positive pressure between the fiberglass and epoxy adhesive. In the present test program this pressure was impossible to apply during specimen manufacture as the cavity was filled with the viscous epoxy adhesive. As pressure is important in removal of micro-disbonds /2/, the absence of a positive pressure during specimen preparation could lead to a decrease in bond strength. Secondly, surface preparation may be different. In the present program the surfaces of the FRP were sanded to remove the resin coat. In the vacuum building the surface of the FRP was ground down to the fiberglass mat as were the previous test specimens.

TABLE 1

Tensile Test Results for Unaged Type A Specimens

Area = 0.81 cm^2

Specimen No.	Load (Kg)	Bond Strength (MPa)
1	170.4	20.7
2	119.2	14.5
3	129.6	15.7
4	86.9	10.5
5	152.7	18.5
6	116.4	14.1
7	59.9	7.3
8	125.9	15.3
9	142.7	17.3

AVERAGE BOND STRENGTH = 14.9 MPa
STANDARD DEVIATION = 3.8 MPa

TABLE 2

Tensile Test Results for Unaged Type B Specimens

Area = 3.2 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	344.2	10.4
2	310.5	9.4
3	175.9	5.3
4	340.9	10.3
5	272.5	8.3
6	broke	---
7	272.5	8.3
8	190.5	5.8
9	209.6	6.4
10	343.8	10.4

AVERAGE BOND STRENGTH = 8.3 MPa
STANDARD DEVIATION = 1.9 MPa

Since surface condition is perhaps the most important parameter in the strength of the bond /2/, a lower bond strength for the present specimens is expected.

As can be seen from the test results, although the load required to break the FRP/Epoxy bond rises with bond area, it does not rise at the same rate resulting in lower stress to break the larger bond areas. This is due to the increase in the percentage of micro-disbonds with bond area causing a weaker bond per unit area /2/ as the area increases. Figure 12 shows a plot of bond strength versus bond area. As can be seen the trend is toward an asymptotic value of bond strength at which level the bond area has no effect. To confirm this trend 30 additional specimens, 10 each of 3 different bond areas were manufactured in the same manner as described previously. Type D had a bond area of 1.6 cm^2 (0.25 in^2) with epoxy dimensions of 1.27 x 1.27 x 2.54 cm. (0.5 x 0.5 x 1.0 in). Type E had a bond area of 4.8 cm^2 (0.75 in^2) with epoxy dimensions of 1.9 x 2.54 x 2.54 cm (0.75 x 1.0 x 1.0 in). Type F had a bond area of 9.7 cm^2 (1.5 in^2) with epoxy dimensions of 2.54 x 3.81 x 2.54 cm (1.0 x 1.5 x 1.0 in). The bond strengths of the Type D, E, and F specimens are listed in Tables 4, 5, and 6. Figure 13 shows the plot of bond strength versus bond area with the new values added. The type D, E, and F specimens were prepared in a similar manner to the type A, B, and C specimens. However, the D, E, and F specimens were prepared with more quality control and therefore were expected to have a smaller range of breaking strengths than the A, B, and C specimens. As expected, the A, B, and C specimens have a wide scatter of bond strength values as indicated by the large values of standard deviation for each of the three bond areas.

Lastly, if the curve shown in Figure 13 is extrapolated to a bond area of 58 cm^2 (9 in^2) which is the bond area between two segments of stiffening rings, the tensile strength has reached an asymptotic value of approximately 3.8 MPa (550 psi). To relate this value to that in the vacuum building, if the same bond strength versus bond area curve is applicable but if a value of 33 MPa, the value obtained from the previous test program, is used as the bond strength for a 1.6 cm^2 bond area, then by shifting the curve in Figure 13, the asymptotic value for a 58 cm^2 bond is 5.6 MPa (815 psi).

5.0 TEST RESULTS FROM SPECIMENS AGED 3 MONTHS

Thirty specimens, 10 each of type A, B, and C, were removed from the aging chambers following three months aging under vacuum and water. The tensile test results from these specimens are shown in Tables 7, 8, and 9. The strain rate for these tests was, as before, 1 mm/minute. All the specimens broke at the FRP/Epoxy adhesive bond.

TABLE 3

Tensile Test Results from Unaged Type C Specimens

Area = 12.9 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	579	4.4
2	712	5.4
3	662	5.0
4	447	3.4
5	329	2.5
6	666	5.1
7	499	3.8
8	832	6.3
9	457	3.5
10	947	7.2

AVERAGE BOND STRENGTH = 4.7 MPa
STANDARD DEVIATION = 1.4 MPa

TABLE 4

Tensile Test Results from Unaged Type D Specimens

Area = 1.6 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	142	8.6
2	125	7.6
3	109	6.6
4	130	7.9
5	125	7.6
6	121	7.3
7	149	9.0
8	108	6.5
9	71	4.3

AVERAGE BOND STRENGTH = 7.3 MPa
STANDARD DEVIATION = 1.3 MPa

TABLE 5

Tensile Test Results from Unaged Type E Specimens

Area = 4.8 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	314	6.4
2	260	5.3
3	294	6.0
4	354	7.2
5	287	5.8
6	303	6.1
7	297	6.0
8	291	5.9
9	261	5.2
10	249	5.0

AVERAGE BOND STRENGTH = 5.9 MPa
STANDARD DEVIATION = 0.58 MPa

TABLE 6

Tensile Test Results from Unaged Type F Specimens

Area = 9.7 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	528	5.3
2	386	3.9
3	524	5.3
4	506	5.1
5	351	3.6
6	483	4.9
7	490	5.0
8	454	4.6
9	391	4.0
10	453	4.6

AVERAGE BOND STRENGTH = 4.6 MPa
STANDARD DEVIATION = 0.59 MPa

The results show a decrease in bond strength with bond area and a large scatter in bond strength for each specimen type. Even though these results seem to indicate a reduction in tensile strength from the unaged specimens, closer examination clearly shows that they are not different statistically. If a confidence level of 90% is chosen, the unaged specimens would have mean bond strengths of 14.9 $\pm$ 6.3 MPa for Type A, 8.3 $\pm$ 3.1 MPa for Type B, and 4.7 $\pm$ 2.3 MPa for Type C. The three month aged specimens have mean bond strengths of 11.9 $\pm$ 3.6 MPa for Type A, 6.8 $\pm$ 6.1 MPa for Type B, and 4.3 $\pm$ 0.9 MPa for Type C. Since the uncertainties for each specimen type overlap, it cannot be said that bond strength degradation has occurred for any bond area type.

The specimens had, at this point in time, unsealed bond perimeters, which, it was felt, lead to water ingress of the bond degrading an unrepresentative percentage of the bond area. As mentioned previously in the report, the rest of the specimens were removed from the aging chambers at this point and the bond perimeters sealed. Since there was water ingress from all sides of the three month aged specimens, the results did not provide the desired information. Therefore, it was proposed to wait for further results to clarify any trends in the bond degradation and not to use the three month aged results as they might be misleading.

6.0 TEST RESULTS OF SPECIMENS AGED FOR 6 MONTHS

Thirty specimens, ten of each type, were removed from the aging chambers after six months of aging under vacuum and water conditions. The tensile test results from these specimens are shown in Tables 10, 11, and 12 for Type A, B, and C specimens respectively. The strain rate for these tests was 1 mm/minute and all specimens broke at the FRP/Epoxy adhesive bond line.

The results show an apparent decrease in bond strength with bond area and a large scatter in bond strength for each specimen type. Using a confidence limit of 90%, the mean bond strengths are 7.5 $\pm$ 4.0 MPa for Type A, 4.0 $\pm$ 1.3 MPa for Type B, and 4.1 $\pm$ 1.1 MPa for Type C. The average bond strength has degraded for each specimen type. This degradation has been most pronounced in the smaller bond area specimens, Types A and B. While Type C specimens show a greater than 10% strength degradation over a six month aging period this is significantly less than the near 50% degradation shown by Types A and B. Since the bond strengths with uncertainties overlap the unaged bond strengths with uncertainties, it cannot be said that any bond strength degradation has occurred in six months aging.

TABLE 7

Tensile Test Results from
3 Month Aged Type A Specimens

Area = 0.8 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	85.0	10.3
2	89.5	10.9
3	146.3	17.8
4	95.5	11.6
5	86.4	10.5
6	113.6	13.8
7	93.6	11.4
8	84.5	10.3
9	92.7	11.2
10	95.0	11.5

AVERAGE BOND STRENGTH = 11.9 MPa
STANDARD DEVIATION = 2.2 MPa

TABLE 8

Tensile Test Results from
3 Month Aged Type B Specimens

Area = 3.2 cm^2

Specimen Number	Load(Kg)	Bond Strength (MPa)
1	477.2	14.5
2	436.3	13.2
3	143.2	4.3
4	136.4	4.1
5	114.5	3.5
6	140.9	4.3
7	172.7	5.2
8	189.1	5.7
9	191.8	5.8
10	225.5	6.8

AVERAGE BOND STRENGTH = 6.8 MPa
STANDARD DEVIATION = 3.7 MPa

TABLE 9

Tensile Test Results from
3 Month Aged Type C Specimens

Area = 12.9 cm^2

Specimen Number	Load (Kg)	Bond Strength (MPa)
1	613.2	4.7
2	542.7	4.1
3	712.7	5.4
4	559.1	4.2
5	572.7	4.3
6	438.2	3.3
7	596.4	4.5
8	596.4	4.5
9	547.7	4.2
10	477.3	3.6

AVERAGE BOND STRENGTH = 4.3 MPa
STANDARD DEVIATION = 0.54 MPa

TABLE 10

Tensile Test Results from
6 Month Aged Type A Specimens

Area = 0.8 cm^2

Specimen Number	Length Exposed to Water (cm)	Load (Kg)	Bond Strength(MPa)
1	0.6	38.6	4.2
2	0.6	84.5	10.3
3	0.6	87.7	10.6
4	0.6	83.6	10.1
5	0.6	broke	----
	AVERAGE BOND STRENGTH = 8.8 MPa		
6	1.3	38.6	4.2
7	1.3	65.4	7.9
8	1.3	60.9	7.4
9	1.3	45.9	5.6
10	1.3	57.3	7.0
	AVERAGE BOND STRENGTH = 6.4 MPa		

AVERAGE BOND STRENGTH FOR ALL SPECIMENS = 7.5 MPa
STANDARD DEVIATION = 2.4 MPa

TABLE 11

Tensile Test Results from
6 Month Aged Type B Specimens

Area = 3.2 cm^2

Specimen Number	Length Exposed to Water (cm)	Load (Kg)	Bond Strength(MPa)
1	1.3	150.0	4.6
2	1.3	76.4	2.3
3	1.3	88.2	2.7
4	1.3	86.8	2.6
5	1.3	139.1	4.2
	AVERAGE BOND STRENGTH = 3.3 MPa		
6	2.5	121.8	3.7
7	2.5	115.9	3.5
8	2.5	127.7	3.9
9	2.5	155.0	4.7
10	2.5	131.4	4.0
	AVERAGE BOND STRENGTH = 4.0 MPa		
	AVERAGE BOND STRENGTH OF ALL SPECIMENS = 3.6 MPa		
	STANDARD DEVIATION = 0.79 MPa		

TABLE 12

Tensile Test Results from
6 Month Aged Type C Specimens

Area = 12.0 cm^2

Specimen Number	Length Exposed to Water (cm)	Load (Kg)	Bond Strength (MPa)
1	2.5	475.5	3.6
2	2.5	660.5	5.0
3	2.5	650.0	4.9
4	2.5	506.8	3.8
5	2.5	530.5	4.0
	AVERAGE BOND STRENGTH = 4.3 MPa		
6	5.1	477.3	3.6
7	5.1	641.8	4.9
8	5.1	542.3	4.1
9	5.1	374.1	2.8
10	5.1	495.5	3.8
	AVERAGE BOND STRENGTH = 3.8 MPa		
	AVERAGE BOND STRENGTH FOR ALL SPECIMENS = 4.1 MPa		
	STANDARD DEVIATION = 0.66 MPa		

7.0 TEST RESULTS FROM SPECIMENS AGED 9 MONTHS

Thirty specimens, ten of each type, were removed from the aging chambers following nine months of aging under water and vacuum and were tested for tensile bond strength. The tensile test results for these specimens are shown in Tables 13, 14, and 15 for specimen types A, B, and C respectively. The strain rate for the tests was 1 mm/minute and all specimens broke at the FRP/Epoxy adhesive interface.

A confidence limit of 90%, as used previously, gives a mean bond strength of 9.7 $\pm$ 3.1 MPa for Type A, 6.8 $\pm$ 2.5 MPa for Type B, and 5.6 $\pm$ 1.1 MPa for Type C specimens. The uncertainties in these results overlap the bond strengths of the unaged specimens. Therefore, based on a statistical analysis of the results, it cannot be determined if any degradation in the bond strengths for any of the three types of bond areas has occurred during the nine month aging period.

8.0 CONCLUSIONS

The major observation following the tensile testing of specimens after zero, 3, 6, and 9 months of aging under vacuum and water conditions is that there appears to be an apparent degradation of tensile bond strength with aging for each bond area if the mean values of bond strength were considered exclusively. Any degradation would be due to the infiltration of water into the bond area thereby increasing the number of micro-disbonds which would lead to a decrease in the bond strength. The second observation is that at this point in time, because of the lack of sufficient data and because the uncertainty in the results are high the degree of degradation and its relationship with bond area cannot be determined and, in fact, it is uncertain that any degradation in the bond strength for any bond area type has actually occurred.

With regards to the effect of length of bond line exposed to the water, it appears that at this time the results are inconclusive. Insufficient data at this point in time is clearly the main factor in the ambiguous quality of the results up to now.

Finally, from the tests on the unaged specimens it was found that the bond strength for the specimens in this study is considerably less than that in the previous study due to specimen preparation. and adhesive formulation. Since the bonds in the Pickering NGS VB and the bonds in the previous study were made using the same techniques and same adhesive formulation, the correct application of the results from the present study would be as an indication of the trend of vacuum building bond strength degradation with aging time, bond area and infiltration perimeter to bond area

TABLE 13

Tensile Test Results from
9 Month Aged Type A Specimens

Area = 0.8 cm^2

Specimen Number	Length Exposed to Water (cm)	Load (Kg)	Bond Strength(MPa)
1	0.6	66.2	8.0
2	0.6	56.7	6.9
3	0.6	59.8	7.3
4	0.6	92.9	11.3
5	0.6	98.0	11.9
	AVERAGE BOND STRENGTH = 9.1 MPa		
6	1.3	103.4	12.5
7	1.3	83.5	10.1
8	1.3	93.4	11.3
9	1.3	67.6	8.1
10	1.3	74.4	9.0
	AVERAGE BOND STRENGTH = 10.2 MPa		
	AVERAGE BOND STRENGTH FOR ALL SPECIMENS = 9.7 MPa		
	STANDARD DEVIATION = 1.9 MPa		

TABLE 14

Tensile Test Results from
9 Month Aged Type B Specimens

Area = 3.2 cm^2

Specimen Number	Length Exposed to Water (cm)	Load(Kg)	Bond Strength (MPa)
1	1.3	137.9	4.2
2	1.3	199.6	6.1
3	1.3	227.7	6.9
4	1.3	286.2	8.7
5	1.3	284.9	8.6
	AVERAGE BOND STRENGTH = 6.9 MPa		
6	2.5	291.7	8.8
7	2.5	301.2	9.1
8	2.5	257.2	7.8
9	2.5	294.8	8.9
10	2.5	267.2	8.1
	AVERAGE BOND STRENGTH = 8.5 MPa		
	AVERAGE FOR ALL SPECIMENS = 7.7 MPa		
	STANDARD DEVIATION = 1.5 MPa		

TABLE 15

Tensile Test Results from
9 Month Aged Type C Specimens

Area = 12.9 cm^2

Specimen Number	Length Exposed to Water (cm)	Load (Kg)	Bond Strength(MPa)
1	2.5	818.3	6.2
2	2.5	777.5	5.9
3	2.5	718.0	5.4
4	2.5	835.5	6.3
5	2.5	712.6	5.4
6	2.5	710.3	5.4
	AVERAGE BOND STRENGTH = 5.8 MPa		
7	5.1	672.2	5.1
8	5.1	680.8	5.2
9	5.1	871.8	6.6
10	5.1	561.5	4.3
	AVERAGE BOND STRENGTH = 5.3 MPa		

AVERAGE BOND STRENGTH OF ALL SPECIMENS = 5.6 MPa
STANDARD DEVIATION = 0.66 MPa

ratio and allow the previous study to indicate the more representative value of the bond strength in the vacuum building.

9.0 RECOMMENDATIONS

Since the aging of the specimens has only occurred for a relatively short period of time until now, it is recommended that the aging and test program be allowed to continue for much longer periods of time. This would provide the volume of data required for a determination of the degree of bond strength degradation under the aging conditions. At the beginning of the test program it was decided to age specimens for a maximum time period of 144 months with testing occurring at 0, 3, 6, 9, 12, 24, 48, 72, 96, and 144 month periods. It is recommended that this schedule continue.

The previous study /1/ showed essentially no aging effects of vacuum and water on the shear strength of FRP bonded with Epoxy

adhesive 'F' over a two year aging period. This data could then be extrapolated over a 20 year station life. Since the station life is well beyond 2000, a shear bond strength degradation test program over a similar time period as the tensile bond strength test program is recommended. The previous study concentrated on a single shear bond area of 3.2 cm^2. To study the effect of bond area on shear strength and the aging effects of vacuum and water on bond area it is recommended that a parallel test program be performed using shear bond test specimens as prescribed by ASTM D 3165 with bond areas of 12.9, 3.2, and 0.8 cm^2. The specimens, 100 of each bond area, will be aged under vacuum and water and tested at times of 0, 3, 6, 9, 12, 24, 48, 96, and 144 months in order to parallel the test program for the tensile specimens.

10.0 ACKNOWLEDGEMENT

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11.0 REFERENCES

1. "Aging of Epoxy and Fiberglass Reinforced Plastic Components - Pickering NGS Dousing System", J.M. Braun, Ontario Hydro Research Division Report 85-135-H, June 18, 1985.

2. **The Science of Adhesive Joints**, J.J. Bikerman, Academic Press, New York, New York, 1961.

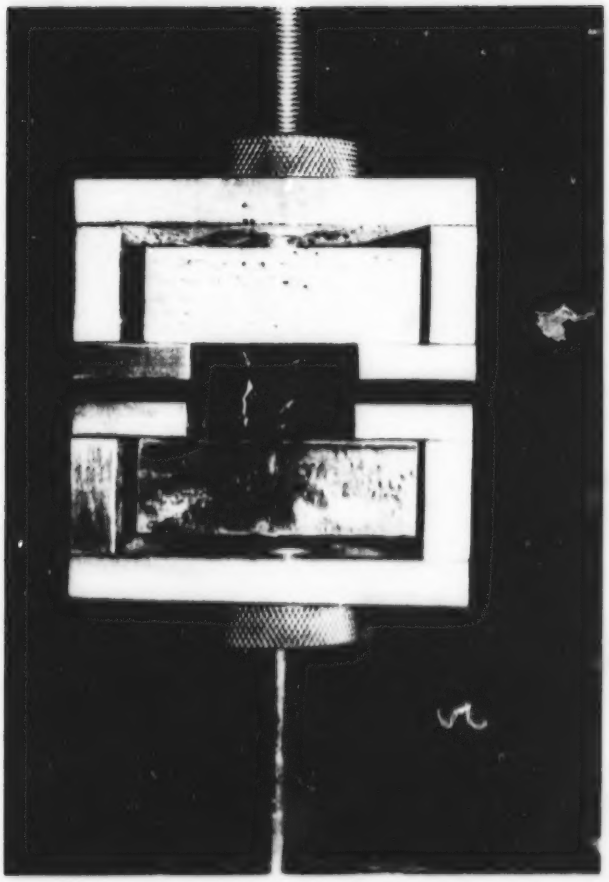

FIGURE 1. SPECIALLY DESIGNED GRIPS FOR LOADING FRP/EPOXY TENSILE SPECIMEN

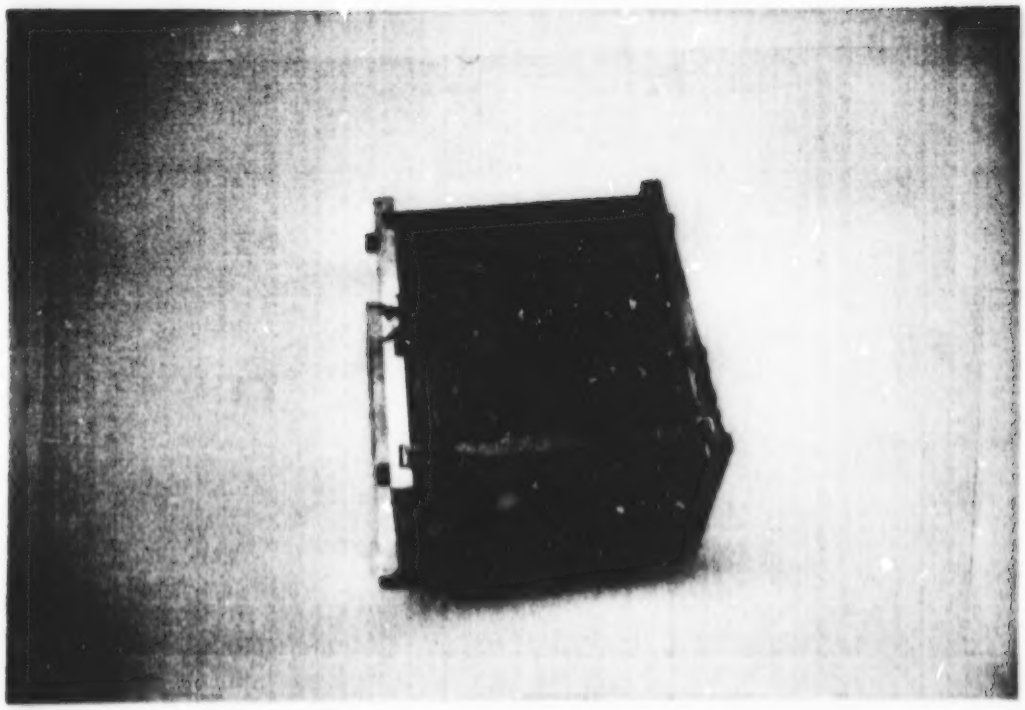

FIGURE 2. MOLD USED FOR MANUFACTURING SPECIMENS

FIGURE 3. TYPE A SPECIMEN

FIGURE 4. TYPE B SPECIMEN

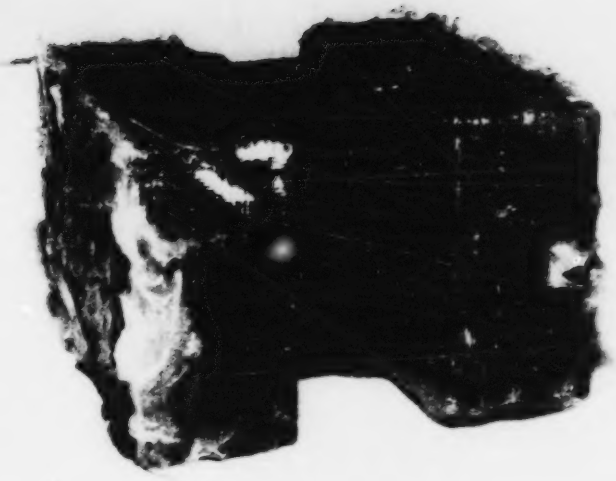

FIGURE 5. TYPE C SPECIMEN

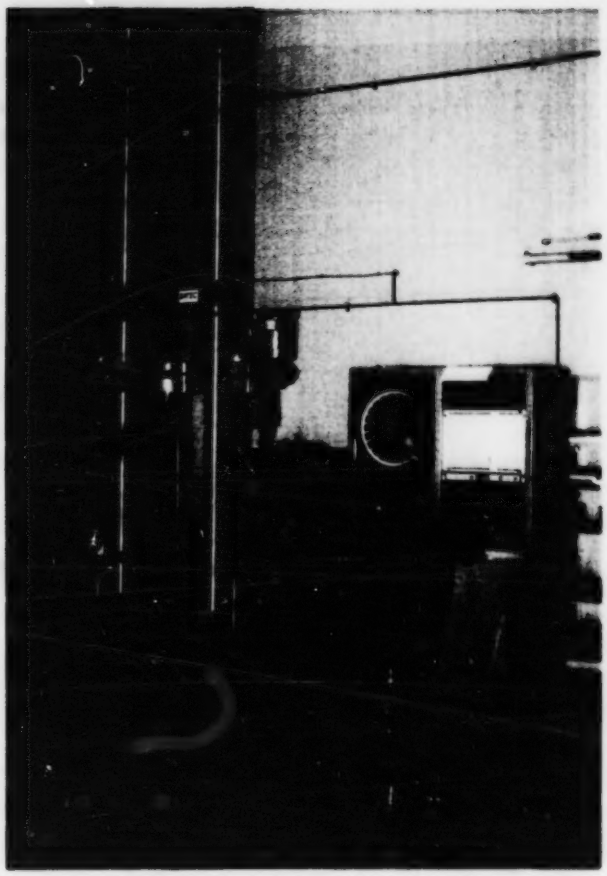

FIGURE 7 SATEC TENSILE TESTING MACHINE USED FOR TESTING TYPE B AND C SPECIMENS

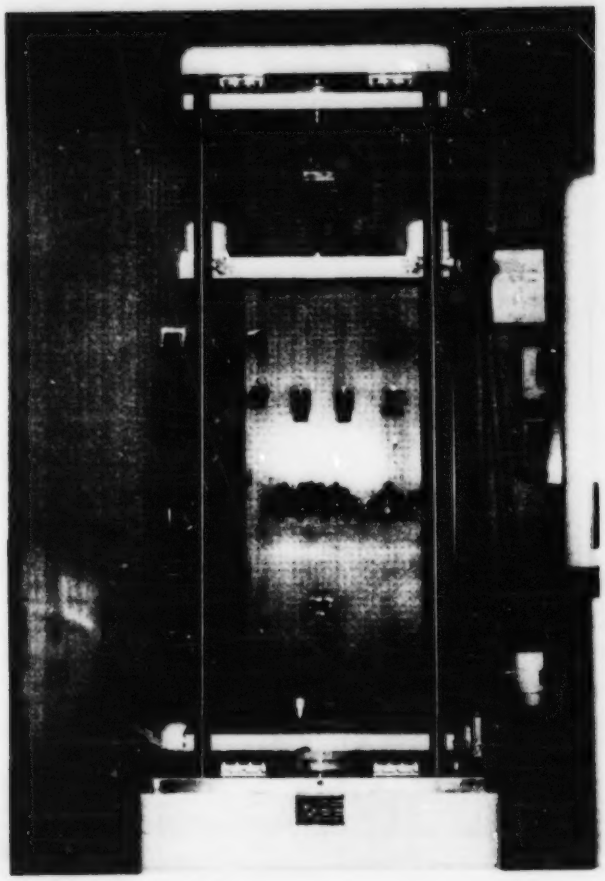

FIGURE 8. BALDWIN TENSILE TESTING MACHINE USED FOR TYPE A SPECIMENS

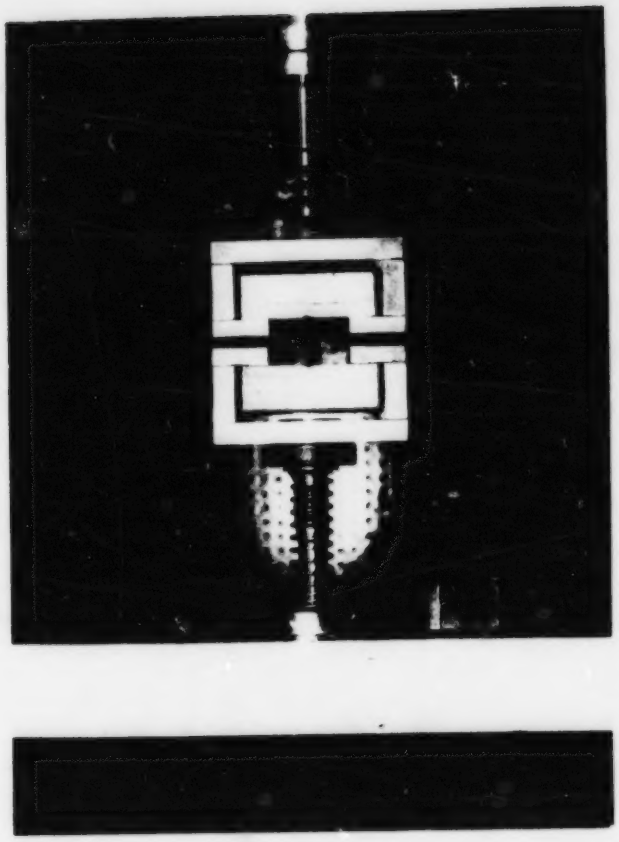

FIGURE 9. TYPE A SPECIMEN UNDERGOING TENSILE TEST ON BALDWIN TESTING MACHINE

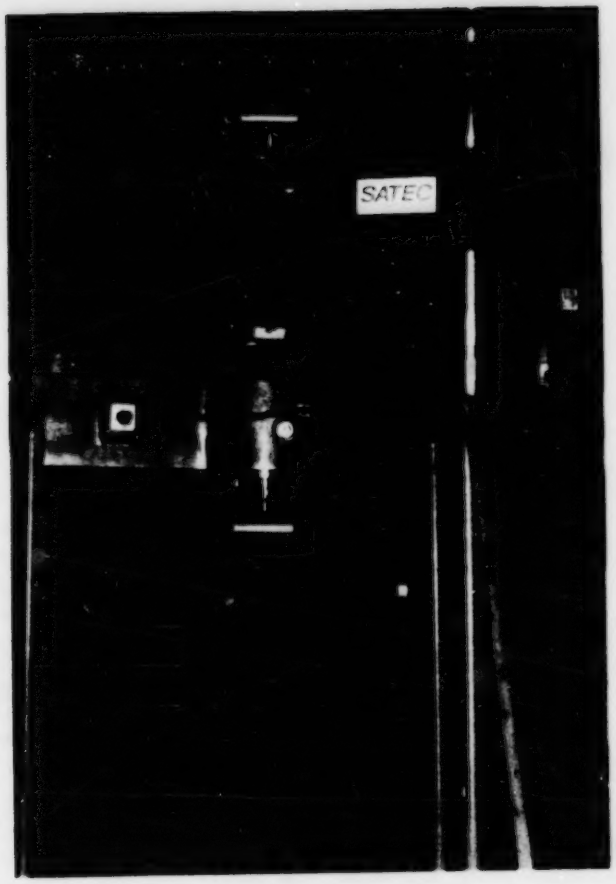

FIGURE 10 TYPE C SPECIMEN UNDERGOING TENSILE TEST ON SATEC TESTING MACHINE

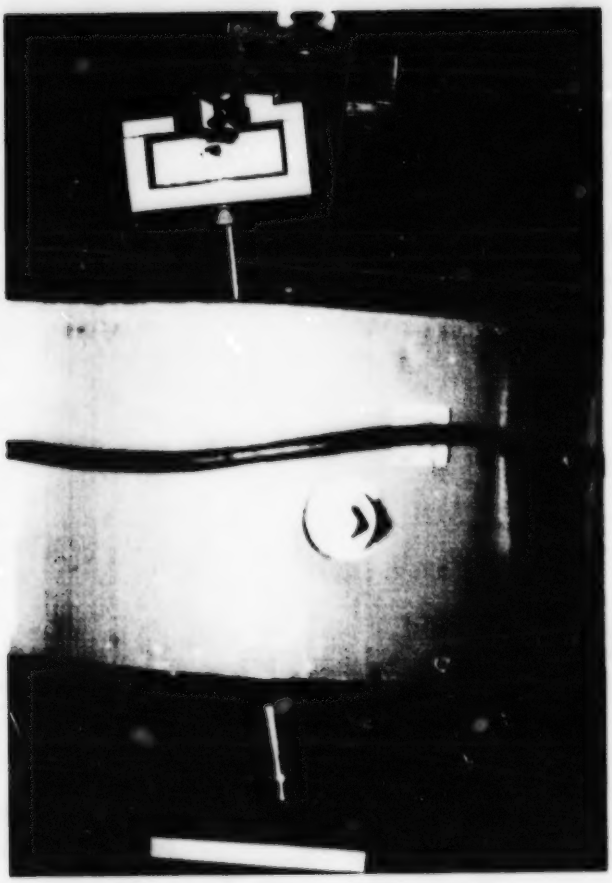

FIGURE 11. SPECIMEN TESTED TO FAILURE - BROKEN AT THE BOND LINE

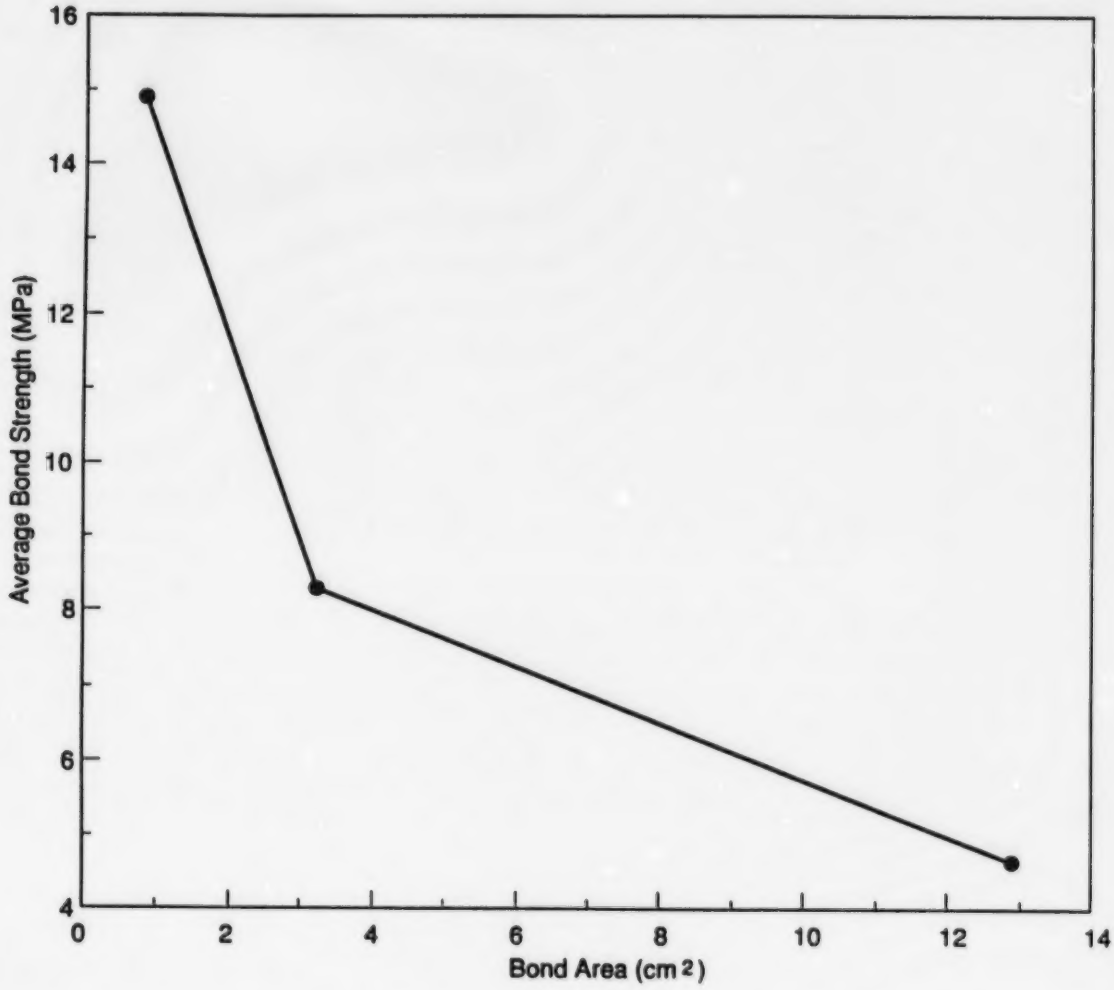

FIGURE 12 AVERAGE BOND STRENGTH VERSUS BOND AREA FOR UNAGED SPECIMENS WITH 3 BOND AREAS

AVERAGE BOND STRENGTH VERSUS BOND AREA

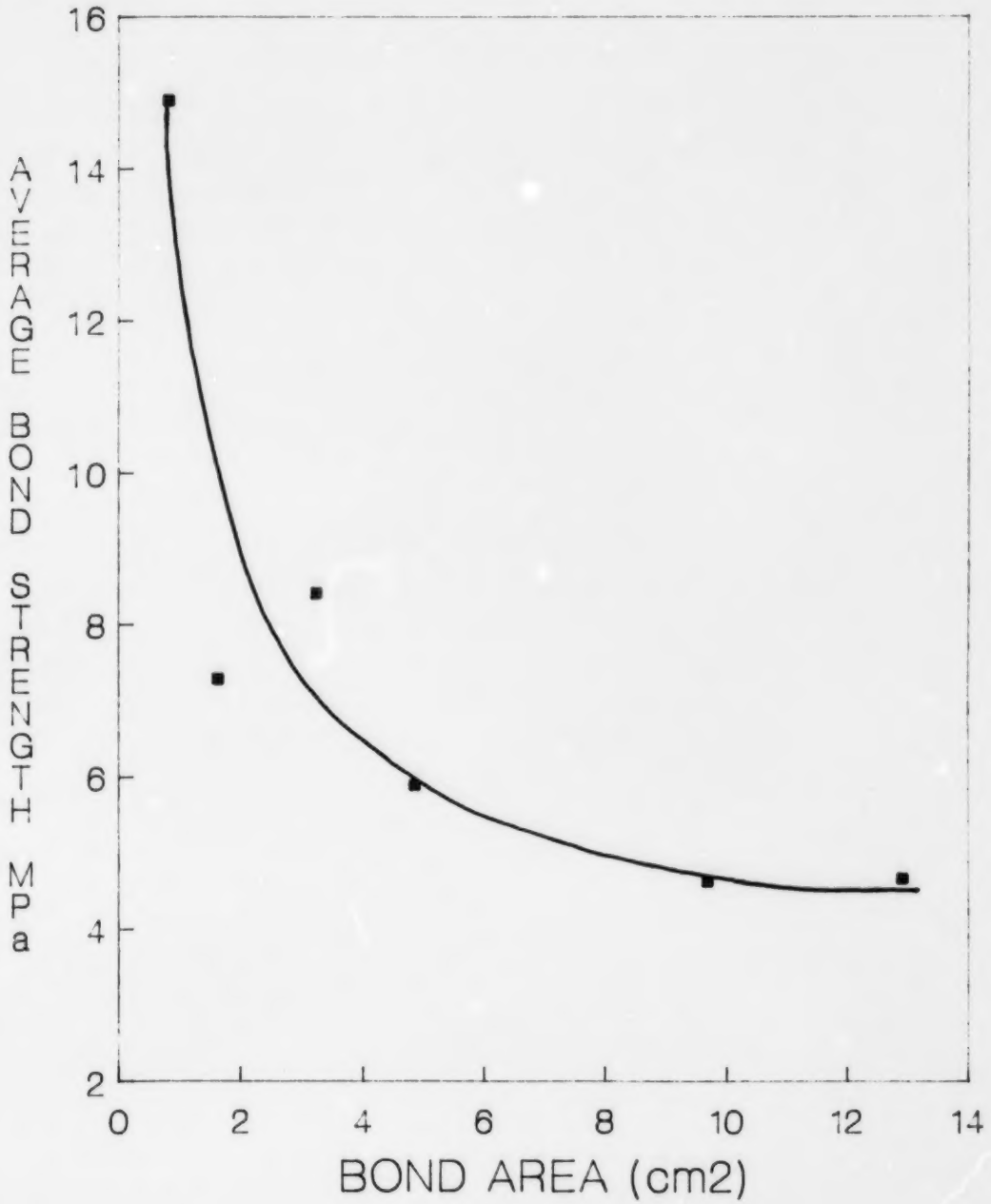

FIGURE 13 AVERAGE BOND STRENGTH VERSUS BOND AREA FOR UNAGED SPECIMENS WITH 6 BOND AREAS